

Qualitative and quantitative analysis of a multilayer polymer film by pyrolysis GC/MS combined with F-Search MPs

[Background] Multilayer films consisting of multiple polymers are used for food packaging materials, providing functions such as gas barrier property, heat sealability, heat resistance, and printability. In the conventional analysis of multilayer films by pyrolysis (Py-) GC/MS, many peaks appeared due to the pyrolysis of multiple polymer components, and it was difficult to identify each polymer component. In this report, the microplastic (MP) analysis method, which is used to analyze mixed samples of 11 polymers, was utilized to the qualitative and quantitative analysis of a multilayer film.

[Experimental] A three-layer film for bread packaging bags of known composition (mass composition ratio; polypropylene (PP) 82.5 : polyethylene (PE) 12.5 : polybutene-1 (PB) 5.0) was used as a model sample for composition analysis. A Py-GC/MS system with a Multi-Shot Pyrolyzer directly connected to the GC inlet was used for measurements. A UAMP column kit, which consists of a pre-column and a main separation column, was used. MP calibration standard (MPCS-SiO2) containing 11 polymers were used for making calibration curves. Furthermore, PB which is not included in the calibration standard was additionally registered in the user library of the F-Search MPs analysis software.

[Results] Software analysis of the pyrograms of the bread packaging bag showed high qualitative match rates of about 90 % for PP, PE, and PB as listed in Table.1. For the quantification of these three polymers, the characteristic pyrolyzates, 2,4-dimethyl-1-heptene (C9'), 1,20-heneicosadiene (C21''), and 2,4-diethyl-1-octene (C12'') were used. The amounts of PP and PE were determined to be 31.9 µg and 8.81 µg, respectively, from the calibration curves obtained using MPCS-SiO2, which corresponds to a mass composition ratio of 69.3 : 19.2 considering a sample mass of 46 µg. However, these determined values were outside the range of the calibration curves, with PP exceeding the upper limit (19.5 µg) and PE below the lower limit (14.3 µg). In addition, PB could not be determined because it was not included in MPCS-SiO2. Therefore, calibration curves were prepared for these three polymers using standard samples, and quantitative values were obtained for each. The resulting mass composition ratios are summarized in Table. 2, which are close to the known composition ratios of the bread packaging bag. The maximum relative standard deviation (RSD) of the quantitative values was 5.2 % (n=3), demonstrating good reproducibility and confirming the effectiveness of the F-Search MPs with a Microplastics Analysis Library for multilayer film analysis.

Table. 1 Match rates of bread packaging bag sample.

Polymer	PP	PE	PB	N66	N6	PMMA	SBR	PC	PS	PET	ABS	PVC
Prob. [%]	98.2	84.9	99.5	67.2	56.8	50.3	50.2	28.1	19.8	13.7	12.1	0.9

Table. 2 Quantification results.

Polymer	PP	PE	PB
Mass composition ratio (formulated value)[%]	82.5	12.5	5.0
Mass composition ratio (quantified value)[%]	82.6	13.8	3.6
RSD (n=3) [%]	1.1	5.2	1.0

Mass composition (normalized to 100)

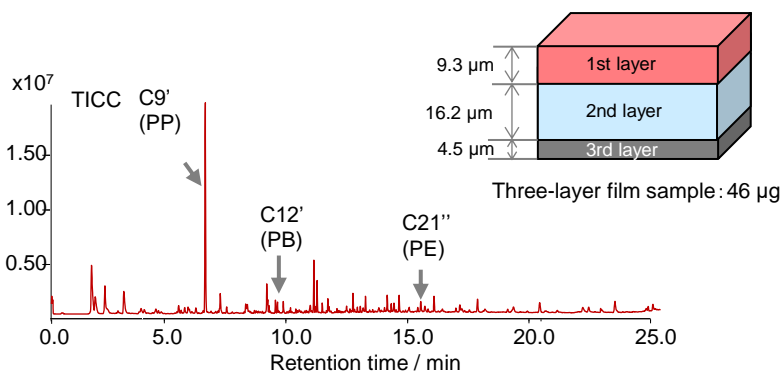


Fig. 1 TICC of bread packaging bag.

Furnace temp.: 600 °C, GC injector temp.: 300 °C, Injector pressure: 75 kPa (const. press.), Split ratio: 1/50
 Pre-column: UA+-50 (50 % diphenyl - 50 % dimethylpolysiloxane; L=1 m, i.d.=0.25 mm, df=1.0 µm),
 Main column: UA+-5 (5 % diphenyl - 95 % dimethylpolysiloxane; L=30 m, i.d.=0.25 mm, df=0.5 µm),
 GC oven: 40 (2 min hold) - 280 °C (20 °C/min, 10 min hold) - 320 °C (40 °C/min 15 min hold),
 GC/MS interface temp.: 300 °C, MS scan range (scan mode): m/z 29-550, Scan rate: 4 scan/s, Film sample amount: 46 µg.

Keywords : Food packaging materials, Mixed polymers, Qualitative analysis, Quantitative analysis

Products used : Multi-Shot Pyrolyzer, UAMP Column Kit, F-Search MPs

Applications : General polymer analysis

Related technical notes :

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